

# *Identifying ancient water availability through phytolith analysis: an experimental approach*

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# Identifying ancient water availability through phytolith analysis: An experimental approach



Emma Jenkins<sup>a,\*</sup>, Khalil Jamjoum<sup>b</sup>, Sameeh Nuimat<sup>c,1</sup>, Richard Stafford<sup>d</sup>,  
Stephen Nortcliff<sup>a</sup>, Steven Mithen<sup>a</sup>

<sup>a</sup> School of Archaeology, Geography and Environmental Science, University of Reading, Whiteknights, Box 277, Reading, RG6 6AB, UK

<sup>b</sup> National Center for Agricultural Research and Extension, Al-Baqah, 19381, Jordan

<sup>c</sup> Ministry of Agriculture, Queen Rania Al Abdullah St 39, 11181, Jordan

<sup>d</sup> Department of Life and Environmental Science, Bournemouth University, Fern Barrow, Poole, BH12 5BB, UK

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This paper is dedicated to the memory of Rob Potter (1950–2014), our Water, Life and Civilisation project colleague who is sadly missed.

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## ABSTRACT

Water management was critical to the development of complex societies but such systems are often difficult, if not impossible, to recognise in the archaeological record, particularly in prehistoric communities when water management began. This is because early irrigation systems are likely to have been ephemeral and as such would no longer be visible in the archaeological record. We conducted a three year crop growing experiment in Jordan to test the hypothesis that phytoliths (opaline silica bodies formed in plants) can be used to detect the level of past water availability and hence be used as a source of information for inferring past water management. Over a three year period we grew native land races of six-row barley (*Hordeum vulgare*) and durum wheat (*Triticum durum*) at three crop growing stations in Jordan with the crops being subjected to different irrigation regimes. Seeds were sown in the autumn and the crops harvested in the spring. The plants were then exported to the University of Reading for phytolith processing. Our results show that while there were unknown factors that influenced phytolith production between years, at the higher levels, the ratio of 'fixed' form phytoliths (those formed as a result of genetically determined silicon uptake) to 'sensitive' form phytoliths (those whose silicon uptake is environmentally controlled) can be used to assess past water availability. Our study is the first large scale experimental project to test this method and take into account multiple variables that can affect phytolith production such as soil composition and chemistry, location, climate and evapotranspiration rates. Results from the cereals grown at two of the crop growing stations, Deir 'Alla and Ramtha, which received between 100 mm and 250 mm rainfall per annum, demonstrate that if the ratio of fixed to sensitive phytolith forms is  $>1$ , the level of past water availability can be predicted with 80% confidence. Results from the crops grown at the other growing station, Kherbet as-Samra, which received less than 100 mm of rainfall per year show that if the ratio of fixed to sensitive forms is  $>0.5$ , the level of past water availability can be predicted with 99% confidence. This demonstrates that phytolith analysis can be used as a method to identify past water availability.

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## 1. Introduction

### 1.1. Irrigation and water management

Irrigation and water management would have been critical to

the development of agriculture in southwest Asia, where the earliest farming communities in the world developed. We know from environmental records that rainfall in this region was often sparse and unreliable during the Holocene making the possibility of crop failure a very real problem for early farmers (see [Rambeau and](#)

\* Corresponding author. Department of Archaeology, Anthropology, and Forensic Science, Bournemouth University, Fern Barrow, Poole, BH12 5BB, UK.

E-mail addresses: [ejenkins@bournemouth.ac.uk](mailto:ejenkins@bournemouth.ac.uk) (E. Jenkins), [khaliljamjoum@yahoo.com](mailto:khaliljamjoum@yahoo.com) (K. Jamjoum), [sameeh.nuimat@iucn.org](mailto:sameeh.nuimat@iucn.org) (S. Nuimat), [rstafford@bournemouth.ac.uk](mailto:rstafford@bournemouth.ac.uk) (R. Stafford), [s.nortcliff@reading.ac.uk](mailto:s.nortcliff@reading.ac.uk) (S. Nortcliff), [s.j.mithen@reading.ac.uk](mailto:s.j.mithen@reading.ac.uk) (S. Mithen).

<sup>1</sup> IUCN, Sweifiyeh, Hasan Baker Al Azazi, Building, no 20, P.O. Box 942230, 11194, Amman, Jordan.

Black, 2011 and Robinson et al., 2011 for a review of the palaeoenvironmental evidence). Documenting when water management began is, however, difficult because early irrigation systems are likely to have been ephemeral and, as such, would no longer be visible in the archaeological record. In an effort to address this, attempts have been made to reconstruct past water availability using organic macrobotanical remains, including studies of grain size (Helback, 1960; Mabry et al., 1996); weed flora (Charles and Hoppé, 2003; Charles et al., 2003; Jones et al., 1995, 2010); and carbon isotopes (Araus and Buxó, 1993; Araus et al., 1997, 1999, 2007; Ferrio et al., 2005; Fiorentino et al., 2008, 2012; Flohr et al., 2011; Heaton et al., 2009; Riehl, 2008; Riehl et al., 2008; Stokes et al., 2011; Voltas et al., 2008; Wallace et al., 2013, 2015). While these methods are useful, their applicability is limited by the need for sufficiently well preserved organic macrobotanical assemblages, both for direct analysis, and for the extraction of isotopes, as well as the expense (in terms of time and money) for isotope analysis.

### 1.2. Phytolith formation

Phytoliths are a more durable form of archaeobotanical evidence which has been used, with some success, as a proxy for past water availability (Madella et al., 2009; Rosen and Weiner, 1994; and Weisskopf et al., 2014). Moreover, they are relatively straightforward and inexpensive to process and analyse. Phytoliths are composed of opaline silica which is taken up as monosilicic acid by plants through their roots into the vascular system during transpiration and then deposited in a solid state as silicon dioxide in inter- and intra-cellular spaces. Two modes of silicon (Si) uptake in plants have been proposed—passive and active, while some species have also been identified as Si rejecters or excluders (Ernst et al., 1995; Jarvis, 1987; Liang et al., 2006; Jones and Handreck, 1967; Ma and Yamaji, 2006; Mayland et al., 1993; Richmond and Sussman, 2003; Takahashi et al., 1990; Walker and Lance, 1991). Both *Triticum aestivum* (Van Der Vorm, 1980; Jarvis, 1987; Rafi and Epstein, 1999; Casey et al., 2003) and *Hordeum vulgare* (Barber and Shone 1966) have been shown to be active Si uptakers. However, both passive and active Si uptake co-exist in many species (Jarvis, 1987; Walker and Lance, 1991; Mayland et al., 1993; Ernst et al., 1995; Liang et al., 2006; Ma and Yamaji, 2006). Active Si uptake is dependent upon external factors such as temperature and external Si concentrations (Liang et al., 2006; Ma and Yamaji, 2006), the latter of which is also dependent on water availability since Si uptake occurs during transpiration. For example, Richardson et al. (1931) found a correlation between water transpired by barley grown in controlled greenhouse conditions and the Si content of the plant (Richardson et al., 1931; cited in Hutton and Norrish, 1974, 204), while Jones and Milne (1963), Jones et al. (1963) and Jones and Handreck (1965) demonstrated that water transpiration affected Si uptake in oats. Similarly, Hutton and Norrish (1974) showed that the amount of silica found in the husks of wheat was proportional to water transpired.

### 1.3. Phytoliths as indicators of past water availability

Madella et al. (2009) proposed that genetically controlled Si uptake leads to the production of what are termed ‘fixed form’ phytoliths. These form in all environmental conditions and are comprised of grass short cells, such as rondels, saddles, bilobates, crosses, trapezoids, and crenates. Conversely, Madella et al. (2009) suggest that environmentally controlled Si uptake leads to the production of ‘sensitive form’ phytoliths, which only occur under favourable environmental conditions for example when there is sufficient water and plant available Si in the soil, these are comprised of grass long cells and stomata (Madella et al., 2009).

Unlike organic macrobotanical remains, phytoliths are inorganic and so do not require specific preservation conditions such as water-logging or charring, although they can be subject to mechanical breakage (Jenkins, 2009) and variable dissolution in alkaline environments (Albert et al., 2006; Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015; Fraysse et al., 2006a, 2006b, 2009; Iler, 1979; Loucaides et al., 2008, 2010). This means that phytoliths are often more ubiquitous on archaeological sites than macrobotanical remains, particularly prehistoric sites, and are therefore critical to our understanding of early agricultural practices.

The potential of phytoliths to inform about past water availability was originally proposed by Rosen and Weiner (1994) and further explored by Madella et al. (2009), Jenkins et al. (2011) and Weisskopf et al. (2014). Rosen and Weiner (1994) hypothesised that the increased level of transpiration in arid and semi-arid regions would affect Si uptake and deposition in cereals to such an extent that it would be discernible in the phytolith assemblage. To explore this they conducted a crop growing experiment in Israel involving dry-farmed and irrigated emmer wheat (*Triticum turgidum* sbsp *dicoccum*) and bread wheat (*T. aestivum*). They analysed phytoliths from the husks of the plants and found that the wheat grown under irrigation not only had a greater yield of phytoliths but also a greater number of conjoined cells (Rosen and Weiner, 1994). However, it was subsequently discovered that conjoined phytolith forms are more prone to mechanical breakage than was previously thought (Jenkins, 2009). This means that while more conjoined cells may form with increased water availability, taphonomic processes in the archaeological record—particularly over a long period of time, can cause these cells to disaggregate making this method problematic unless your phytolith assemblage has remained undisturbed through time.

Madella et al. (2009) explored the possibility of using phytoliths as an indicator of past water availability using a method that does not rely on conjoined forms. Their study involved five different cereals: bread wheat (*T. aestivum*), emmer wheat (*T. dicoccum*), spelt wheat (*T. spelta*), two row barley (*Hordeum vulgare*) and six row barley (*H. distichon*). These were grown under two different climatic regimes: Middle Eastern and Northern European. Middle Eastern climatic conditions were simulated using a growing chamber and included both wet and dry regimes. The wet regime involved keeping the pots under water holding capacity, defined as “the amount of water that the soil can ‘hold’ above oven-dry” (Madella et al., 2009; page 34) with water being administered on a daily basis, while the dry regime was irrigated to 50% of the water holding capacity. For the Northern European climatic conditions, the plants were grown in open fields in Cambridge which has an annual rainfall of around 600 mm. Phytoliths from the leaves of all plants were analysed, while phytoliths from the culms (stems) were also analysed for emmer and spelt wheat (Madella et al., 2009).

Madella et al. (2009) looked at the ratio of fixed to sensitive phytolith forms and the percent of sensitive forms in the cereals they analysed. They showed that in leaves there was an increase in the ratio of fixed to sensitive forms under the wet regime for bread wheat, emmer wheat and two-row barley. For the spelt wheat and six-row barley there was an overlap in the values, while in the culms (stems) no significant difference was found in the ratios for either emmer or spelt wheat.

The results of Madella et al. (2009) are valuable and suggest that the ratio of fixed to sensitive phytolith forms can be used as a proxy for water availability. However, their work, as with most initial experimental research, had some limitations which largely relate to the experimental design and statistical analyses. Firstly, the plants grown under the Middle Eastern climatic regime were cultivated in pots in a greenhouse rather than in open fields. These plants would

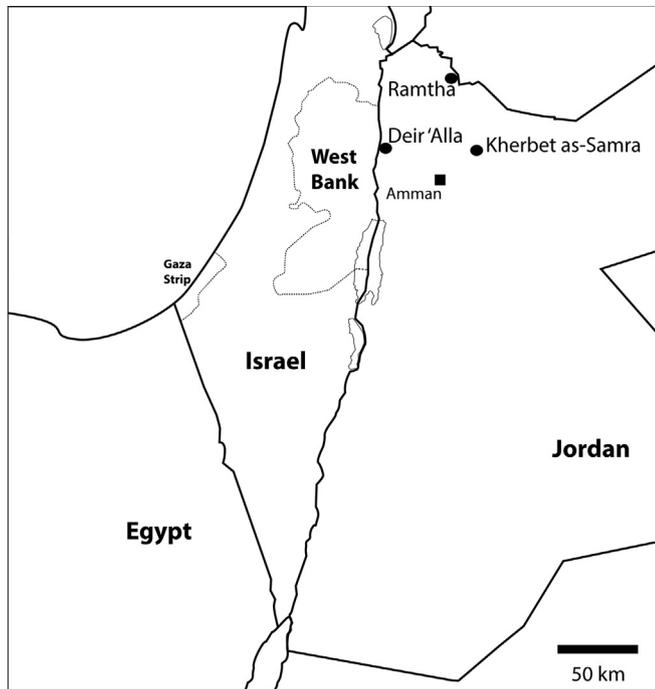


Fig. 1. Location of crop growing stations used in this study.

have received little competition for water and nutrients; they would presumably have been weed free allowing for maximum Si uptake, potentially impacting upon the resulting phytolith assemblage. Secondly, greenhouses increase humidity levels and, unless

dehumidifiers were used, which is not made clear in the report, it is probable that the level of humidity in the greenhouse would have been higher than natural for a Middle Eastern arid environment, affecting evapotranspiration rates. Thirdly, no information was provided regarding the amount of water given to the plants, and how this relates to their known crop water requirements. Fourthly, the soil Si levels were not measured and so we do not know if Si levels in the sediments used for the pot experiments were the same as those in the sediments in the open fields. Lastly, limited statistical analysis was undertaken in this study so the robustness and significance of the results remains unclear.

Another study by Weisskopf et al. (2014) used the method outlined by Madella et al. (2009) to identify the growing conditions of rice during the Neolithic in the Lower Yangtze valley, China. Weisskopf et al. (2014) analysed phytolith samples from modern rice fields which were categorised according to water availability as: 1) dry, rain-fed and margin of wetland; 2) very wet, in standing water throughout most of the growing season; or 3) intermediate (Weisskopf et al., 2014). They calculated the percent of sensitive to fixed forms found in the samples that were presumed to have originated from both the rice and the accompanying Poaceae weeds (Weisskopf et al., 2014). They found that the percent of sensitive to fixed forms could be used to monitor wetter and drier growing conditions, which indicated changes in rice cultivation from flooded and drained fields to intensively irrigated paddies (Weisskopf et al., 2014). Their results demonstrate the promise of the method but the research did not involve any additional method testing.

## 2. Aims

The aim of our research is to further explore the validity of using phytoliths to indicate past water availability by:

Table 1

Location, altitude and slope of site for the three crop growing stations used in this study: Kherbet as-Samra, Ramtha and Deir 'Alla (after Carr, 2009).

	Kherbet as-Samra	Ramtha	Deir 'Alla
Location (latitude and longitude)	N32° 08-890 E36° 08-710	N32° 34 E36° 1	N32° 11-483 E035° 37-167
Altitude (metres)	567 m above sea level	500–600 m above sea level	–192 m below sea level
Slope (at soil sampling localities)	<3°	<3°	<3°
Precipitation average (mm/year)	150	300–350	250



Fig. 2. Harvesting one of the 5 m plots at Kherbet as Samra.

**Table 2**  
Amount of applied irrigation (mm per year) by crop, year and growing station.

Irrigation regime	Deir 'Alla	Ramtha	Kherbet as-Samra
Year 1 (2005–6) barley			
0%	25.0	25	25.0
80%	74.1	101.5	124.2
100%	92.6	126.8	155.2
120%	111.1	152.2	186.3
Year 1 (2005–6) wheat			
0%	25.0	25.0	25.0
80%	91.2	150.7	176.0
100%	114.0	188.4	220.1
120%	136.8	226.1	264.1
Year 2 (2006–7) wheat and barley			
0%	25.0	25.0	25.0
40%	28.4	41.0	41.3
80%	56.8	82.0	82.5
100%	71.0	102.5	103.2
120%	85.2	123.0	123.8
Year 3 (2007–8) wheat and barley			
0%	25.0	25.0	25.0
40%	88.9	60.2	79.2
80%	177.8	120.4	158.4
100%	222.3	150.5	198.1
120%	266.7	180.6	237.7

- 1) determining whether the ratio of short (fixed) forms to long (sensitive) forms from husks of durum wheat (*T. durum*) and six-row hulled barley (*H. vulgare*) is affected by water availability in the same way as it is for leaves and stems (Madella et al., 2009)
- 2) considering the affect that other variables could have on phytolith formation, namely rainfall, evapotranspiration rates and soil composition/chemistry.

While preliminary results for these experiments have been published by Jenkins et al. (2011), this is the first time that a full statistical analysis of them is available providing readers with a definitive outline of the advantages and limitations of the method.

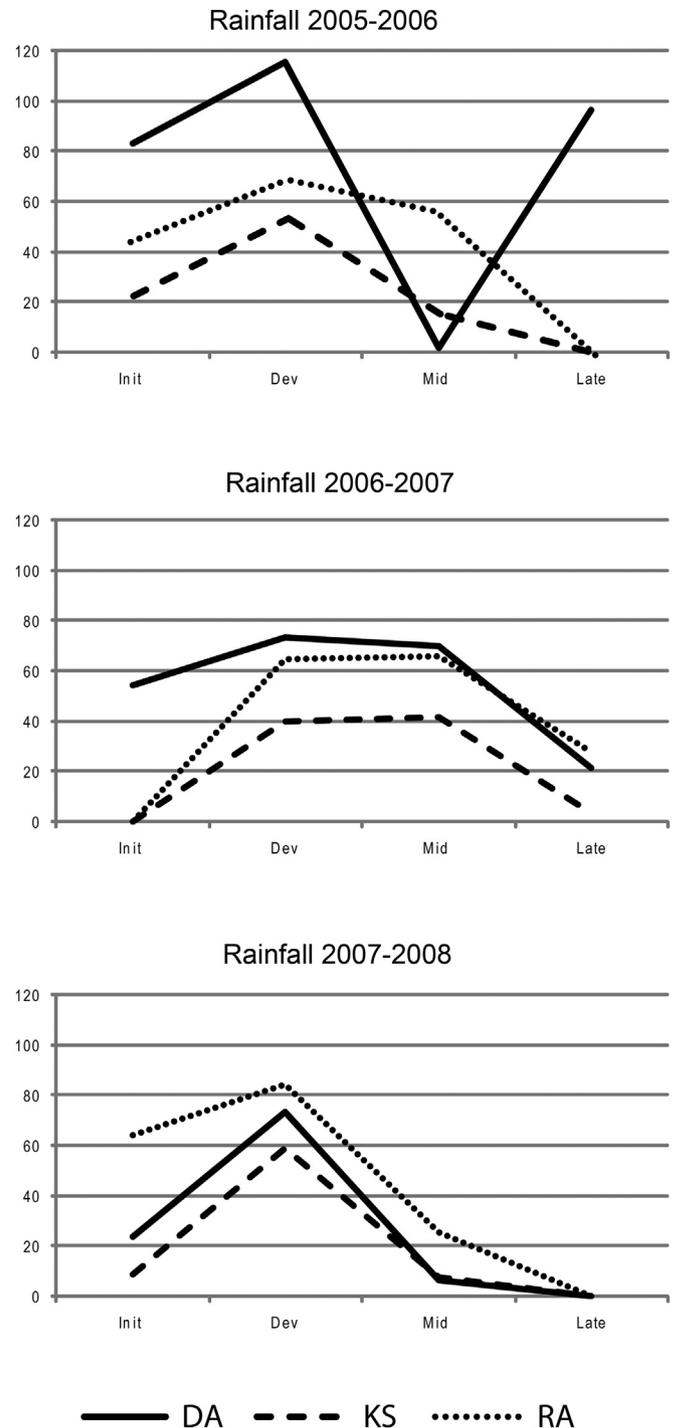
### 3. Material and methods

#### 3.1. Field materials and methods

Two crops were grown for phytolith analysis, both of which were native land races: durum wheat (*T. durum*) (ASCAD 65) and six row hulled barley (*H. vulgare*) (ASCAD 176). These were grown for three years, 2005–2008, at three different crop growing stations in Jordan: (1) Kherbet as-Samra, which is on the Jordanian Plateau to the northeast of Amman; (2) Ramtha, which is in the north of Jordan, 5 km from the Syrian border; and (3) Deir 'Alla which is in the Jordan valley (Fig. 1; Table 1; Mithen et al., 2008).

Each experimental plot measured 5 m by 5 m and was surrounded by a soil bund with a 1.5 m separation from the adjacent plot (Fig. 2). In the first season, four different irrigation regimes were employed on the basis of known crop water requirements estimated using Class A – Pan evaporation readings (Allen et al., 1998): (1) no irrigation- 0% of crop water requirements; (2) under irrigated-80% of crop water requirements; (3) irrigated-100% of crop water requirements; and (4) over irrigated-120% of crop water requirements. In the second and third seasons an additional under-irrigated plot was added which was given 40% of crop water requirements (Table 2).

Daily rainfall and evaporation levels were accounted for when calculating the amount of irrigated water to apply in order to meet the specified conditions for each regime. Water was provided by a drip irrigation system with a 60 cm spacing between each water pipe and a 40 cm spacing between the drippers on the pipes. Each



**Fig. 3.** Irrigation in mm by crop development stage. Crop development stages (given on the x axis) follow the Food and Agricultural Organisation convention of Initial (Init.), Crop development (Dev.), Mid-Season (Mid.) and Late. Please note the difference in rainfall for Kherbet as Samra and Ramtha in 2005–2006 in this figure as compared to Jenkins et al., 2011. In the former publication there is an error whereby Kherbet as Samra is mislabelled as Ramtha and Ramtha is mislabelled as Kherbet as-Samra.

irrigation plot had eight lines. The water used for irrigation was treated waste water at Kherbet as-Samra and Ramtha and a mixture of treated waste water and fresh water at Deir 'Alla. The water used was within the Jordanian standards for the irrigation of restricted crops (Carr, 2009). All plots, including the unirrigated ones, were given 25 mm of water after sowing to encourage germination.

Ensuring that crops were irrigated to the correct levels was not

without problems. For example, if there was a heavy rain storm just after the plants had been irrigated, water could not retrospectively be taken away from the plants meaning that occasionally some plots received too much water. Fig. 3 provides information about rainfall.

Crops were planted in November and harvested in May. In the first year of experimentation barley was harvested before wheat at Ramtha and Deir 'Alla. However, due to practicalities, the two crops were harvested at the same time during the second and third year of experimentation at all sites. As a result of this decision, irrigation levels for the barley and wheat were the same for all plots and sites in the second and third year of the experiment. No pesticides or fertilisers were used on the crops and the plots were not weeded. Bird attack was an ongoing problem at Deir 'Alla and Ramtha, with some plots having to be covered with mesh for protection (Table 3). As the mesh was not applied until the plants were approaching maturity, evapotranspiration rates were unaffected for the majority of the growing season.

During harvesting, a grid system was laid diagonally across the plots and plants were collected from six 50 cm intervals: 0–50 cm, 50–100 cm, 100–150 cm, 150–200 cm, 200–250 cm, and 250–300 cm. This was done to avoid edge effect (where plants on the edge of plots receive more water and nutrients from the ground because of the decreased competition). Plants were placed inside paper bags after collection and soil samples were taken from each plot at three different depths: 0–5 cm, 5–25 cm and >25 cm.

### 3.2. Laboratory materials and methods

Soil samples were characterised and analysed at the Department of Soil Science, University of Reading (Carr, 2009). Soil samples were also taken after the first and last year of experimentation

**Table 3**  
Record of mesh coverage by year, crop species and crop growing station.

		Year 1	Year 2	Year 3
Deir 'Alla	barley	X	✓	X
	wheat	✓	✓	✓
Ramtha	barley	✓	✓	✓
	wheat	✓	✓	X
Kherbet as'Samra	barley	X	X	X
	wheat	X	X	X



**Fig. 4.** Images of fixed forms or short cells (cork cells) labelled as A and sensitive or long cells (elongate dendriforms) labelled as B from the experimentally grown wheat in this study.

to test for extractable soil Si. Phytolith extraction followed the dry ashing method (see Jenkins et al., 2011; page 357 for a full description of methods). Weight percent of phytoliths was calculated by expressing the weight of phytoliths to original plant matter processed (phytolith weight % = weight of phytoliths/weight of plant matter processed \* 100). This is useful for determining the level of Si uptake and resulting phytoliths in the plant.

Only the husks of the plants were processed and cork cells (fixed forms) and elongate dendriforms (sensitive forms) (Fig. 4) were counted using a Leica DME at x 400. A decision was made not to include papillae phytoliths in the count because we observed that

**Table 4**

Extractable soil Si levels from the experimental crop growing plots shown as mg Si kg<sup>-1</sup> air-dry soil and as mg SiO<sub>2</sub> kg<sup>-1</sup> dry soil.

Year	Crop	Site	Irrigation	Soil concentration mg/kg AD soil	
				Si	SiO <sub>2</sub>
2006	Barley	KS	0	251	539
2006	Barley	KS	80	205	439
2006	Barley	KS	100	221	473
2006	Barley	KS	120	229	490
2006	Barley	DA	0	268	575
2006	Barley	DA	80	311	666
2006	Barley	DA	100	268	575
2006	Barley	DA	120	264	566
2006	Barley	RA	0	301	646
2006	Barley	RA	80	312	669
2006	Barley	RA	100	333	713
2006	Barley	RA	120	311	666
2006	Wheat	KS	0	205	440
2006	Wheat	KS	80	223	479
2006	Wheat	KS	100	229	491
2006	Wheat	KS	120	230	492
2006	Wheat	DA	0	291	623
2006	Wheat	DA	80	270	579
2006	Wheat	DA	100	269	577
2006	Wheat	DA	120	265	567
2006	Wheat	RA	0	333	715
2006	Wheat	RA	80	322	691
2006	Wheat	RA	100	335	719
2006	Wheat	RA	120	357	765
2008	Barley	KS	0	301	644
2008	Barley	KS	40	269	576
2008	Barley	KS	80	245	526
2008	Barley	KS	100	243	520
2008	Barley	KS	120	241	516
2008	Barley	DA	0	316	677
2008	Barley	DA	40	292	625
2008	Barley	DA	80	273	584
2008	Barley	DA	100	268	574
2008	Barley	DA	120	257	550
2008	Barley	RA	0	412	883
2008	Barley	RA	40	393	842
2008	Barley	RA	80	372	797
2008	Barley	RA	100	358	768
2008	Barley	RA	120	352	754
2008	Wheat	KS	0	294	631
2008	Wheat	KS	40	267	572
2008	Wheat	KS	80	259	555
2008	Wheat	KS	100	261	560
2008	Wheat	KS	120	262	562
2008	Wheat	DA	0	299	640
2008	Wheat	DA	40	260	557
2008	Wheat	DA	80	274	586
2008	Wheat	DA	100	247	530
2008	Wheat	DA	120	292	626
2008	Wheat	RA	0	400	857
2008	Wheat	RA	40	361	774
2008	Wheat	RA	80	380	815
2008	Wheat	RA	100	356	764
2008	Wheat	RA	120	334	715

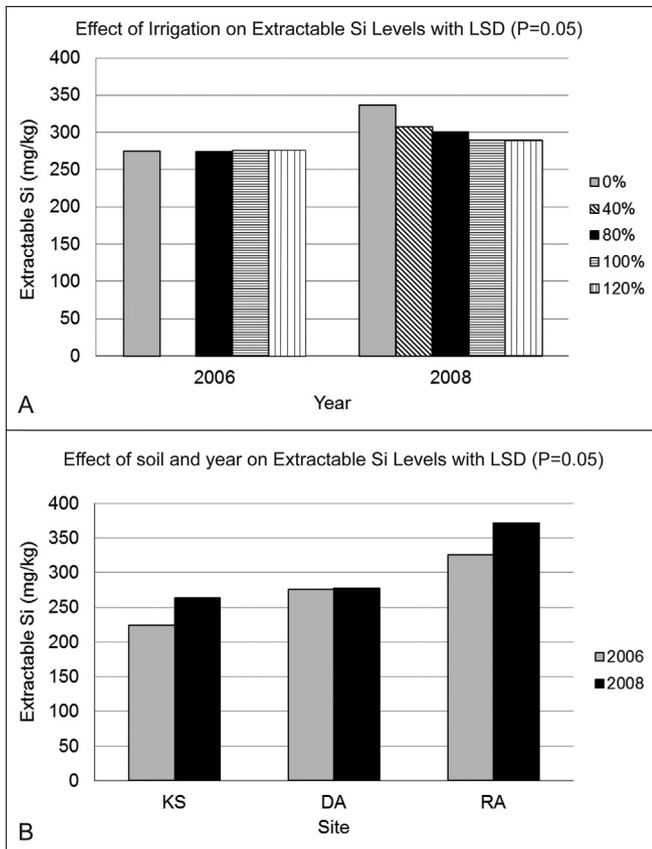


Fig. 5. Extractable soil silicon levels in the experimental growing plots at the end of the growing seasons in 2006 and 2008.

these forms were not well-preserved in all of our samples. This is in accord with results from previous studies that have made similar observations about the poor preservation of papillae phytoliths in soil samples (Alexandre et al., 1997; Barboni et al., 1999; Albert et al., 2006; Finne et al., 2010; Novello et al., 2012). Ten slides per plot were counted in the first year and five slides in the second and third years. In total, 264 wheat samples were counted, producing 63,135 phytolith forms, and 245 barley samples producing 90,855 phytolith forms.

### 3.3. Statistical methods

Statistical analysis was performed in R 3.0.2 (R core team, 2013). The ratio of fixed to sensitive forms was used as a dependent variable, and was square root transformed to ensure it met the assumptions of the parametric tests used (Underwood, 1997). A multiple regression was performed with 'site', 'plant species' and 'year' as fixed factors (categorical variables) in the analysis, and 'irrigation treatment', as a proportion of full irrigation, as a continuous variable. The minimum adequate model was then calculated using backwards stepwise reduction of the model (following the procedures in Crawley, 2007; page 335). To calculate the importance of irrigation treatment in the analysis, as compared to the other variables, the proportion of the variability explained by each factor was calculated using the 'relaimpo' package (Grömping, 2006), providing an  $r^2$  value for each factor in the model, and thus providing information on how much variability in the ratio of fixed to sensitive forms could be explained by the manipulated levels of irrigation.

The effect of extractable soil Si levels on the ratio of fixed to

sensitive cells was also investigated. Since data for Si were not available for 2006–2007, an analysis identical to the multiple regression above was conducted but with the inclusion of Si as an additional independent variable, and the removal of all data for the years 2006–2007.

Using this dataset with 2006–2007 removed, we also investigated the role of measured environmental variables ('initial, developmental, mid-season and late-season rainfall' and 'evaporation rates' and 'parts per million of clay in the soil', 'pH levels', 'plant species', 'extractable soil Si' and 'level of irrigation'). Since these environmental variables should alter between site and between years, these additional variables replaced the former categorical variables of 'site' and 'year' in the model.

Next, to assess if irrigation treatment could be predicted from the ratio of fixed to sensitive forms, a binomial regression was performed using irrigation as either present (80, 100 and 120% of optimal level) or absent (no irrigation). For clarity all data referring to 40% irrigation levels were discarded for this analysis, as this level of irrigation was thought to create uncertainty in the analysis. It was removed *a priori* to any analysis being conducted. This analysis was performed across all sites combined, then individually for each site.

## 4. Results

Results of the extractable soil Si demonstrated that Si levels were higher in 2008 compared to 2006 for the Kherbet as-Samra and Ramtha sites, but not for Deir 'Alla (Table 4; Fig. 5a). There were no significant differences associated with the crop. Irrigation rate had no significant effect in 2006, but in 2008, Si levels were significantly higher in the non-irrigated treatment (0%) than in the irrigated treatments (Table 4; Fig. 5a). The largest differences in Si levels were found between sites (Table 4; Fig. 5b). Extractable soil Si increased in the order Kherbet as-Samra < Deir 'Alla < Ramtha and the differences between sites were found to be highly significant using an ANOVA test.

Fig. 6 shows that the 0% irrigated wheat plots generally have lower phytolith weight percents than the irrigated wheat plots, and that Deir 'Alla most frequently has the highest weight percent. The most striking observation that can be made from the mean phytolith weight percent for barley (Fig. 7) is that the values for barley are much lower than for wheat. It is also clear that the 0% irrigated plots and the 100% irrigated plots see an overall increase in weight percent over time.

Results of the calculation of mean percent of sensitive forms from wheat (Fig. 8) demonstrate that, with the exception of the first growing season at Ramtha, the mean percent of sensitive forms in the irrigated samples is higher than in the unirrigated ones. However, there is an overlap in values for the irrigated and unirrigated samples from Deir 'Alla. It is also clear that there is a general decrease in the mean percent of sensitive forms through time. The results from the analysis of percent sensitive forms in barley (Fig. 9) show that the mean percent of sensitive forms in the irrigated samples is greater than for the unirrigated ones. There is also a decrease through time in the mean percent of sensitive forms for barley, particularly between the second and third years (see Tables 5 and 6 for the mean percent of elongate dendriforms to cork cells for the different years, sites and irrigation regimes).

The multiple regression analysis produced a significant result explaining around 43% in the variability of difference in ratios of fixed to sensitive forms ( $F_{6,496} = 64.22$ ;  $p < 0.0001$ ;  $\text{adj } r^2 = 0.430$ ). Stepwise reduction (based on AIC) did not remove any of the explanatory variables in the model. The greatest proportion of variability was explained by the variable 'year' (21.1%); with 'irrigation level' explaining 13.4%; 'plant species' explaining 7.2%; and

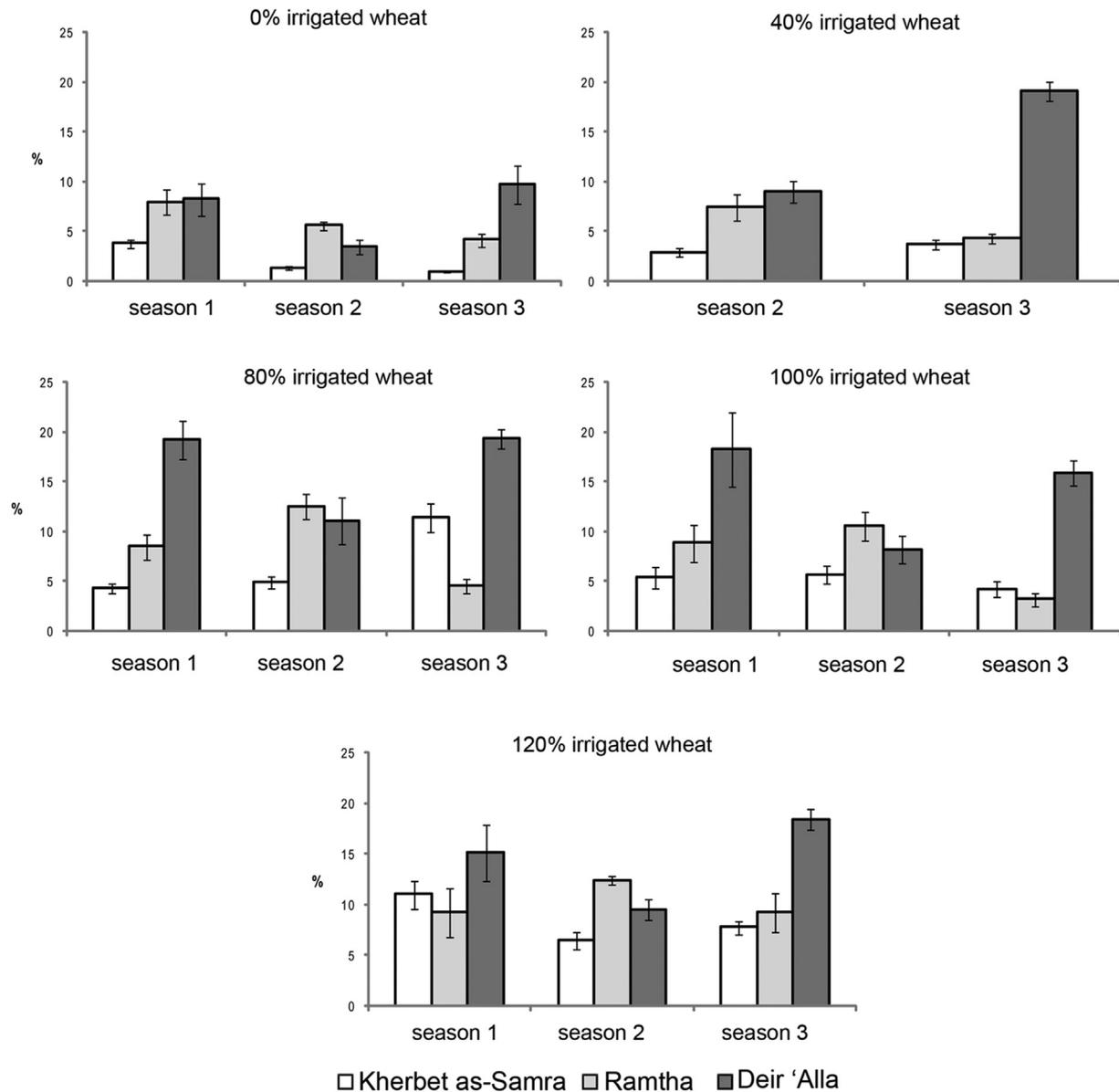


Fig. 6. Weight percent of phytoliths to original plant matter processed for wheat.

'site' explaining just 2.0%.

The inclusion of extractable soil Si in the regression model did create a model with significantly more explanatory power than without ( $F_{6,348} = 49.96$ ;  $p < 0.0001$ ;  $\text{adj } r^2 = 0.454$ ; ANOVA comparing Si model to non-Si model  $p = 0.0043$ ; variability explained by factor - year = 20.8%; irrigation level = 11.4%; site = 5.6%; silicon 5.3%, plant species = 3.2%). Si itself, however, did not explain a large proportion of the variability in the ratio of fixed to sensitive cells.

When examined using environmental variables rather than 'year' and 'site' the reduced model showed slightly higher explanatory power ( $F_{10,492} = 44.6$ ,  $p < 0.0001$ ;  $\text{adj } r^2 = 0.476$ ). The level of irrigation was the factor explaining the most variability in the data (12.9%), with rain in the initial, developmental and middle stages explaining 2.5%, 7.8% and 1.6% respectively (total rainfall explains 11.9%). The species of plant (7.4%), pH (1.4%) and evaporation in the different stages (all  $< 5\%$ ) were the only other significant factors in the reduced model, with Si not being retained.

Logistic regression showed that across all sites, the ratio of fixed

to sensitive cells explained around 15.7% of the variability in levels of irrigation (pseudo  $r^2 = 0.157$ ;  $p < 0.001$ ). However, where the ratio was  $> 1$ , irrigation could be correctly assumed with 80% confidence (Fig. 10a) demonstrating that at the higher ratios phytoliths can be used effectively to determine past water availability. At Kherbet as-Samra, the explanatory power was much greater (pseudo  $r^2 = 0.450$ ;  $p < 0.001$ ) with a ratio of  $> 0.5$  meaning irrigation could be predicted with  $> 99\%$  confidence (Fig. 10b). The other sites showed more variability (Deir Alla - pseudo  $r^2 = 0.122$ ;  $p < 0.001$ ; Ramtha - pseudo  $r^2 = 0.119$ ;  $p < 0.001$ ) and less predictive power (Fig. 10c and b).

## 5. Discussion

The results from the extractable soil Si analysis were surprising because we had expected to see a decrease in the level of extractable soil Si over time whereas the reverse was the case. This was because the experimentally grown plants took up Si from the soil which was not returned in phytolith form because the crops were

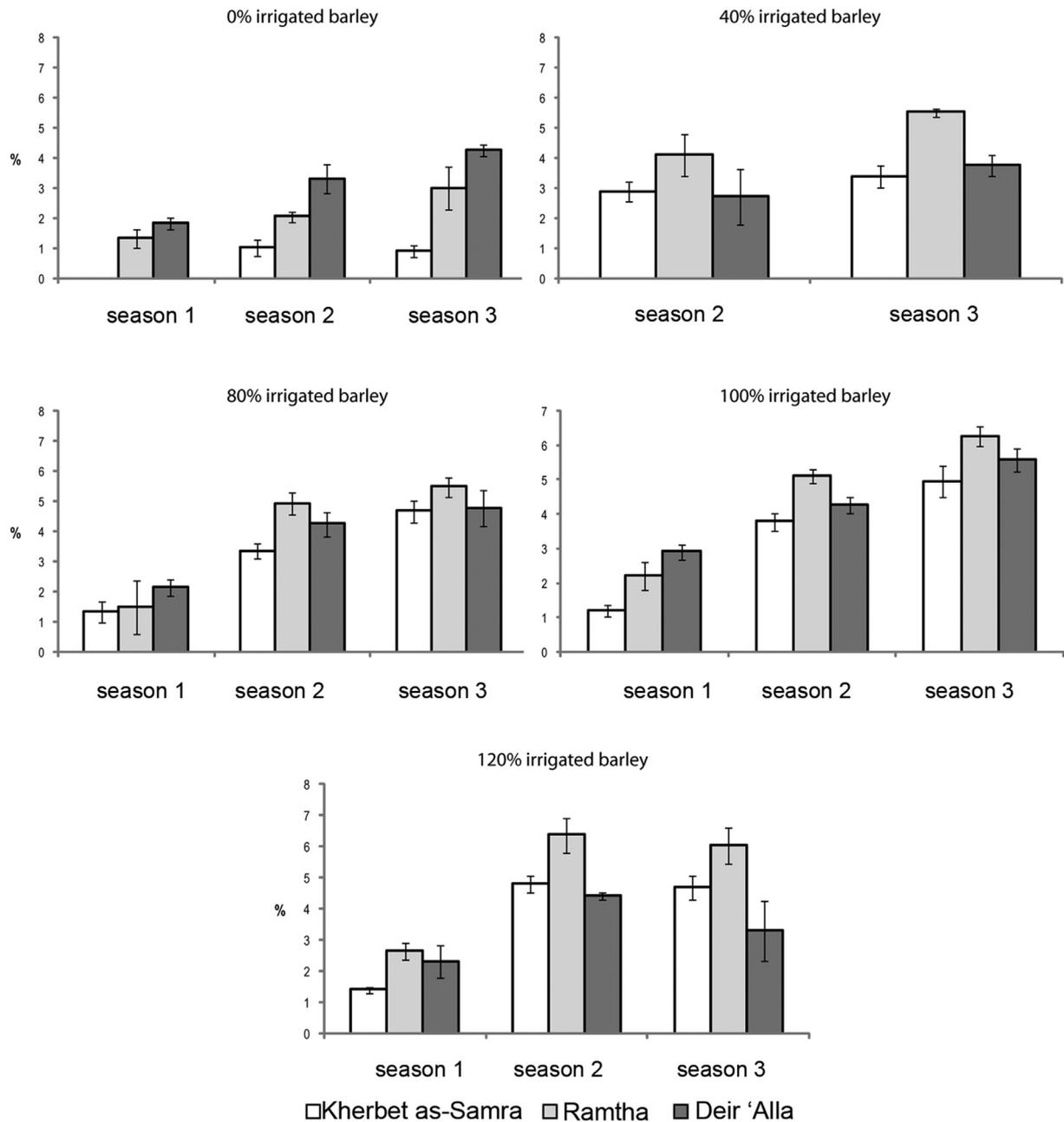


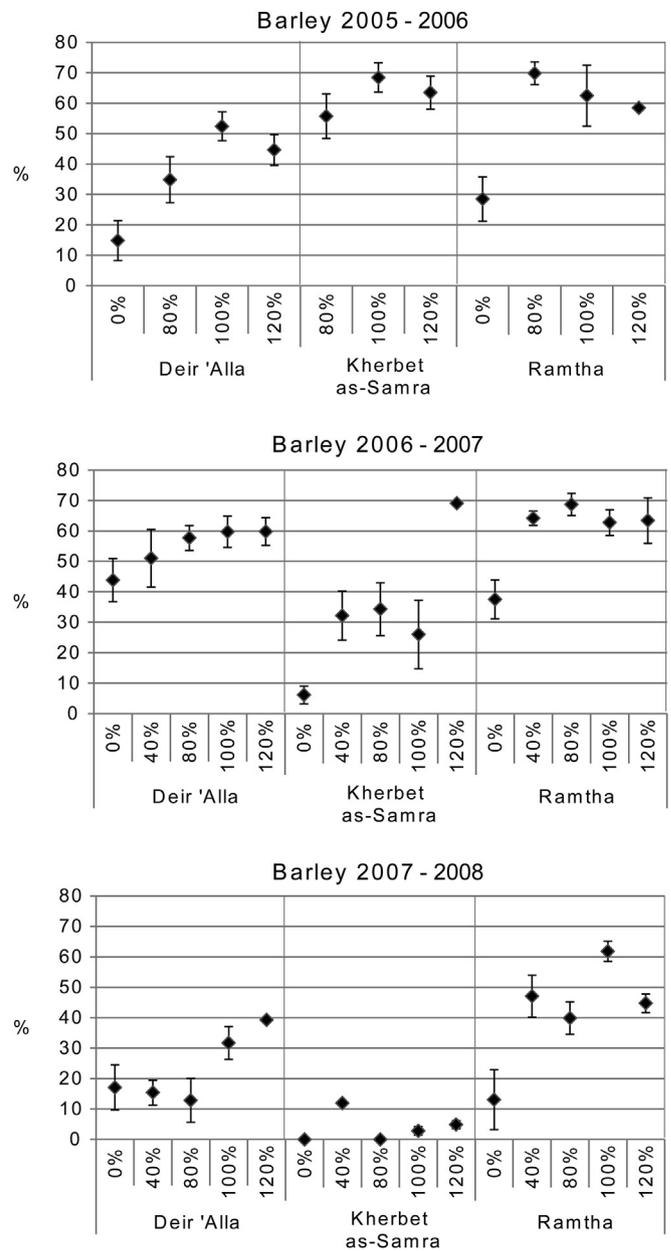
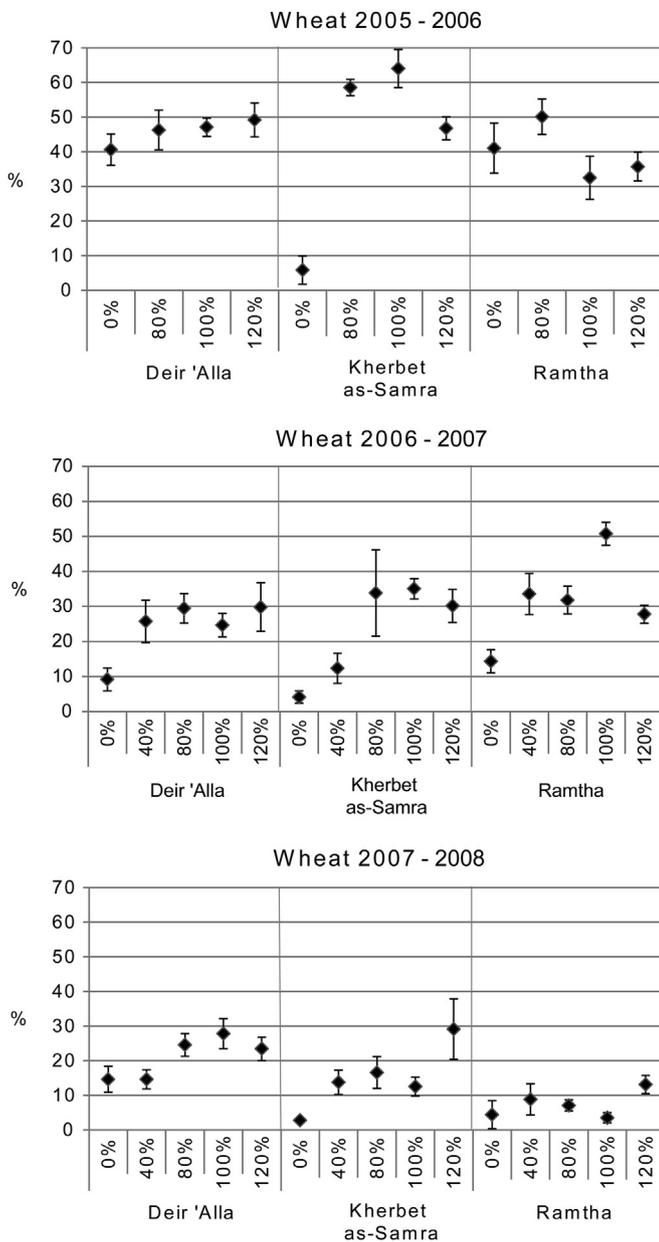
Fig. 7. Weight percent of phytoliths to original plant matter processed for barley.

harvested and removed from the growing sites. Si could not have been added to the soil through the irrigation water because the results show that the greatest rise in Si levels was in the unirrigated plots. The most plausible explanation for this anomaly is that the clay mineral fraction was being washed down through the soil profile by the irrigation water, removing the clay silicates from the rooting zone of the plants. This would explain why the unirrigated samples have more extractable soil Si than the irrigated ones.

The results from the analysis of the weight percent demonstrate that wheat is more prolific in its uptake of Si than barley. This finding is in accord with research conducted by Ma and Takahashi (2002) who compared the Si uptake of six different gramineous species including wheat and barley. They found that while wheat was the second most prolific uptaker of Si after rice, barley took up the least amount. Similarly, Tsartsidou et al. (2007) compared the

number of phytoliths per gram of dry material from a variety of plants collected from sites in Israel and Greece including *H. vulgare* and *T. aestivum*. While the number per gram from the inflorescences of both the *H. vulgare* and *T. aestivum* was the same, results from the whole plant showed that *T. aestivum* had over twice as many phytoliths per gram as the *H. vulgare*. Results from the Greek sites showed that *T. aestivum* had a greater number per gram than the *H. vulgare* for all samples.

The high explanatory power of year as reflected in the multiple regression analysis matches the decrease in mean sensitive forms by year (Figs. 8 and 9). One explanation for this could be rainfall particularly for Deir 'Alla and Kherbet as-Samra. While rainfall was taken into account when calculating irrigation levels, the method was not always perfect as discussed above due to occasional erratic and heavy rainfall. A slight decrease in rainfall occurred between



**Fig. 8.** Percent of sensitive forms or long cells (elongate dendriforms) in samples from the experimentally grown wheat husks.

**Fig. 9.** Percent of sensitive forms or long cells (elongate dendriforms) in samples from the experimentally grown barley husks.

growing years for Kherbet as Samra (year one = 91.7 mm, year two = 85.1 mm, year three = 75.4 mm), while there was a dramatic decrease in levels at Deir 'Alla (year one = 297.8 mm, year two = 220.6 mm, year three = 103.4 mm). Ramtha did not experience a decrease in rainfall over time but saw a slight decrease in the second year compared to the first and third years (year one = 171.1 mm, year two = 159 mm, year three = 174.2 mm).

This hypothesis is supported by the results from the reduced model when 'year' and 'site' were removed as fixed variables but rainfall was included as an independent variable. Analysis found that 'irrigation' was the variable with the highest explanatory power (12.9%), followed by 'rainfall' (11.9%) indicating that water availability is a significant factor in the increased ratio of fixed to sensitive forms. It does, however, also demonstrate that there are factors other than rainfall that have contributed to the high explanatory power of 'year' in the multiple regression analysis.

Our results also indicate that while some variation in the ratio of

fixed to sensitive forms could be explained by differences in species, both durum wheat and six-row barley produce more sensitive forms when irrigation is increased. This contradicts the research of Madella et al. (2009) who found that there was a significant overlap in the percentage of sensitive forms in both of the six row barley samples they analysed and suggest their result could be attributable to a laboratory/experimental error; our research supports this suggestion.

The logistic regression undertaken in our study demonstrated that at the higher levels the ratio of fixed to sensitive forms could be used to identify water availability. For example, if rainfall is between 100 and 250 mm per annum, as was the case at two of the crop-stations (Ramtha and Deir 'Alla), a ratio of fixed to sensitive forms of >1 meant that irrigation could be predicted with 80% confidence. However, when rainfall was less than 100 mm per annum, as was the case at the other crop-growing station (Kherbet

**Table 5**

Mean percent with standard deviation of elongate dendriforms to cork cells by sites, regime and year from the wheat samples.

Site/regime/year	Wheat						
	n slides	Elongate dendriform	Mean	SD	Cork cell	Mean	SD
<b>2005–2006</b>							
DA 0%	10	1277	128	29.7	1868	187	61.0
DA 80%	10	785	79	25.7	911	91	36.8
DA 100%	10	648	65	13.6	728	73	14.6
DA 120%	10	767	77	26.7	793	79	25.2
KS 0%	10	385	39	57.3	6228	623	544.6
KS 80%	10	1519	152	32.4	1076	108	21.9
KS 100%	10	1075	108	49.8	604	60	20.6
KS 120%	10	830	83	17.9	945	95	38.0
RA 0%	10	924	92	24.6	1328	133	46.1
RA 80%	10	954	95	21.6	949	95	25.2
RA 100%	10	738	80	34.1	1535	154	35.1
RA 120%	10	925	93	15.1	1668	167	55.6
<b>2006–2007</b>							
DA 0%	5	139	28	15.5	1368	274	75.1
DA 40%	5	258	52	31.8	745	149	23.5
DA 80%	5	258	52	13.0	617	123	27.4
DA 100%	5	284	57	11.7	867	173	95.4
DA 120%	5	261	52	20.9	614	123	28.7
KS 0%	5	62	12	9.2	1425	285	37.2
KS 40%	5	427	85	34.8	3019	604	271.5
KS 80%	5	497	99	27.6	971	194	153.9
KS 100%	5	433	87	17.3	802	160	55.0
KS 120%	5	380	76	17.4	879	176	55.8
RA 0%	5	325	65	12.1	1937	387	238.3
RA 40%	5	442	88	26.1	874	175	64.4
RA 80%	5	260	52	16.9	557	111	26.2
RA 100%	5	449	90	21.9	435	87	36.4
RA 120%	5	294	59	12.4	764	153	59.3
<b>2007–2008</b>							
DA0%	5	143	29	13.5	835	167	42.21
DA 40%	5	133	27	10.4	779	156	31.23
DA 80%	5	179	36	11.2	550	110	17.31
DA 100%	5	212	42	14.4	550	110	18.64
DA 120%	5	219	44	22.6	716	143	75.16
KS 0%	1	6	6	N/A	210	210	N/A
KS 40%	5	137	27	8.8	858	172	43.04
KS 80%	5	130	26	22.2	654	131	77.43
KS 100%	5	141	28	12.7	988	198	38.60
KS 120%	5	206	41	19.4	502	100	31.48
RA 0%	5	39	8	7.3	850	170	52.69
RA 40%	5	74	15	14.5	767	153	29.44
RA 80%	5	62	12	5.0	818	164	41.05
RA 100%	5	38	8	5.4	1047	209	47.75
RA 120%	3	68	23	9.7	450	150	19.16

as-Samra), a ratio of fixed to sensitive forms of >0.5 meant that irrigation could be predicted with 99% confidence.

Furthermore, our results in addition to those of Madella et al. (2009) and Weisskopf et al. (2014) show that there is a growing body of evidence to suggest that the change in ratio of fixed to sensitive forms as a result of water availability may be a pan-gramineae phenomenon. If this is the case, then this method has a global application and is not restricted to southwest Asian cereals. Further research is needed to confirm this hypothesis but with at least seven crop species, and their accompanying weeds (in the case of rice in the research of Weisskopf et al., 2014), showing positive results this method has great potential.

We do, however, acknowledge that as archaeologists we are frequently dealing with archaeobotanical assemblages that may have been grown over different years and under different environmental and irrigation conditions. As archaeologists it is frequently the case that our understanding of the formation and taphonomy of the sites and samples we study is limited. As such, we do not advocate the use of this method to try and identify specific changes in environmental and irrigation practices within a

**Table 6**

Mean percent with standard deviation of elongate dendriforms to cork cells by sites, regime and year from the barley samples.

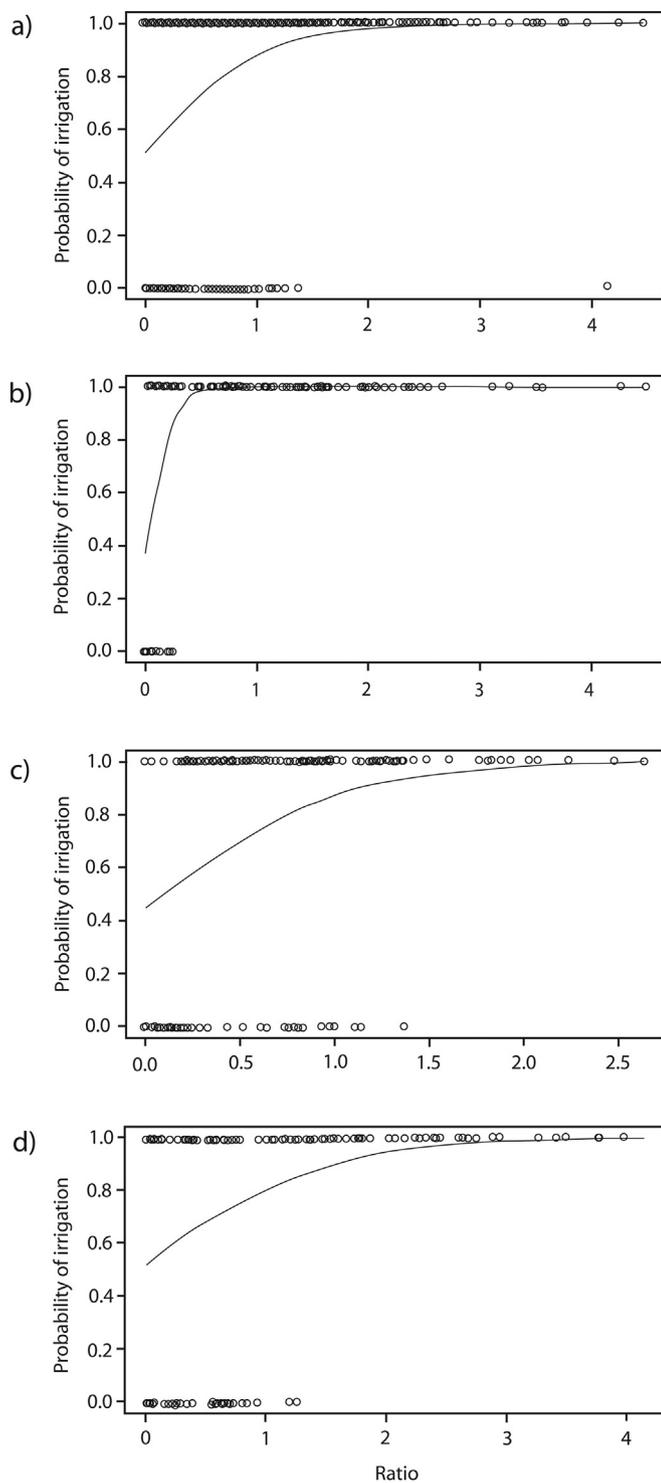
Site/regime/year	Barley						
	n slides	Elongate dendriform	Mean	SD	Cork cell	Mean	SD
<b>2005–2006</b>							
DA 0%	10	679	68	47.8	3900	390	190.2
DA 80%	10	1306	131	51.4	2443	244	99.8
DA 100%	10	1724	172	38.3	1567	157	56.3
DA 120%	10	1443	144	45.1	1793	179	78.7
KS 0%	0	0	N/A	N/A	0	N/A	N/A
KS 80%	10	1640	164	59.9	1305	131	41.1
KS 100%	10	3956	396	40.3	1826	183	95.6
KS 120%	10	2210	221	66.2	1272	127	28.3
RA 0%	9	858	95	49.1	2154	239	101.7
RA 80%	10	2149	215	39.7	930	93	26.6
RA 100%	10	1966	197	71.2	1181	118	68.8
RA 120%	9	1716	191	67.1	1219	135	41.6
<b>2006–2007</b>							
DA 0%	5	506	101	26.5	649	130	51.7
DA 40%	3	352	117	53.2	345	115	29.5
DA 80%	5	693	139	22.0	508	102	21.7
DA 100%	5	776	155	26.4	524	105	31.2
DA 120%	5	696	139	25.6	468	94	20.8
KS 0%	5	77	15	14.6	1186	237	16.7
KS 40%	5	382	76	40.3	806	161	49.0
KS 80%	5	314	63	28.7	602	120	72.5
KS 100%	5	211	42	44.0	602	120	36.1
KS 120%	5	784	157	12.6	351	70	6.6
RA 0%	5	390	78	26.5	651	130	31.4
RA 40%	5	792	158	15.9	442	88	16.2
RA 80%	5	801	160	16.1	365	73	18.9
RA 100%	5	658	132	38.9	391	78	13.8
RA 120%	5	681	136	35.8	393	79	30.8
<b>2007–2008</b>							
DA0%	5	166	33	32.8	806	161	41.9
DA 40%	4	127	32	17.3	698	175	23.4
DA 80%	5	83	17	22.7	563	113	43.7
DA 100%	4	180	45	18.6	388	97	37.7
DA 120%	1	77	77	0.0	119	119	0.0
KS 0%	1	0	0	0.0	239	239	0.0
KS 40%	5	81	16	10.8	600	120	27.6
KS 80%	4	0	0	0.0	107	27	11.9
KS 100%	3	4	2	1.4	138	69	19.8
KS 120%	5	16	3	3.6	311	62	47.7
RA 0%	5	110	22	31.8	735	147	46.3
RA 40%	5	382	76	28.3	430	86	19.3
RA 80%	5	305	61	16.1	460	92	21.2
RA 100%	5	532	106	10.4	329	66	14.4
RA 120%	5	401	80	9.7	495	99	15.3

site over the short term. Instead this method is useful for identifying broad trends at sites with a long history of occupation, or between sites within the same region which experience similar environmental conditions, to identify whether irrigation was practiced at some sites as opposed to others.

In summary, this method can provide a strong indication of past water availability, and of irrigation if it is known that the study region was arid or semi-arid during the period of occupation, when the ratios of fixed to sensitive forms are high. We would suggest, however, that further research is necessary to explore how differential dissolution of phytolith forms in alkaline sediments could affect this method (Albert et al., 2006; Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015; Frayse et al., 2006a, 2006b, 2009; Iler, 1979; Loucaides et al., 2008, 2010).

## 6. Conclusion

We experimentally grew durum wheat and six-row barley at three crop growing stations for three years in Jordan to assess the



**Fig. 10.** Logistic regression predictions for irrigation based on the ratio of short to long cells. (a) all sites combined, (b) Kherbet as Samra, (c) Deir Alla, (d) Ramtha. Points represent raw data and line represents prediction likelihood based on logistic regression, typically a ratio  $>1$  predicts irrigation has occurred with 80% certainty. Note different horizontal scale at site DA, due to no data for high ratios.

effectiveness of using phytoliths to identify past water management. We employed different levels of irrigation to the experimental plots and used the fixed to sensitive phytolith ratio method (Madella et al., 2009; Weisskopf et al., 2014). Logistic regression analysis found that when rainfall was between 100 mm and

250 mm per annum a ratio of fixed to sensitive forms of  $>1$  meant that irrigation could be predicted with 80% confidence. When rainfall was less than 100 mm, the explanatory power was even greater with a ratio of  $>0.5$  meaning irrigation could be predicted with 99% confidence. These findings confirm the validity of using phytoliths to identify past water availability at the three sites we looked at. Further experimental phytolith research involving an array of Gramineae species grown in a range of geographical locations could potentially create a robust method by which past water availability could be identified at archaeological sites globally. Additional research is also needed to explore how differential dissolution of phytoliths in alkaline environments may affect the ratio of fixed to sensitive forms (Albert et al., 2006; Cabanes et al., 2011; Cabanes and Shahack-Gross, 2015; Fraysse et al., 2006a, 2006b, 2009; Iler, 1979; Loucaides et al., 2008, 2010).

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### References

- Albert, R.M., Bamford, M.K., Cabanes, D., 2006. Taphonomy of phytoliths and macroplants in different soils from Olduvai Gorge (Tanzania) and the application to Plio-Pleistocene palaeoanthropological samples. *Quat. Int.* 148, 78–94.
- Alexandre, A., Meunier, J.-D., Lézine, A.-M., Vincens, A., Schwartz, D., 1997. Phytoliths: indicators of grassland dynamics during the late Holocene in intertropical Africa. *Palaeogeogr. Palaeoclimatol. Palaeoecol.* 136, 213–229.
- Allen, R.G., Pereira, L.S., Raes, D., Smith, M., 1998. *Crop Evapotranspiration - Guidelines for Computing Crop Water Requirements*. Food and Agriculture of The United Nations, Rome. Irrigation and drainage paper 56.
- Araus, J., Buxó, R., 1993. Changes in carbon isotope discrimination in grain cereals from the North-Western Mediterranean basin during the past seven millennia. *Aust. J. Plant Physiology* 20, 117–128.
- Araus, J., Febrero, A., Buxó, R., Rodríguez-Ariza, M., Molina, F., Camalich, M., Martín, D., Voltas, J., 1997. Identification of ancient irrigation practices based on the carbon isotope discrimination of plant seeds: a case study from the south-East Iberian Peninsula. *J. Archaeol. Sci.* 24, 729–740.
- Araus, J., Febrero, A., Catala, M., Molist, M., Voltas, J., Romagosa, I., 1999. Crop water availability in early agriculture: evidence from carbon isotope discrimination of seeds from a tenth millennium BP site on the euphrates. *Glob. Change Biol.* 5, 201–212.
- Araus, J.L., Ferrio, J.P., Buxó, R., Voltas, J., 2007. The historical perspective of dryland agriculture: lessons learned from 10,000 years of wheat cultivation. *J. Exp. Bot.* 58, 131–145.
- Barber, D.A., Shone, M.G.T., 1966. The absorption of silica from Aqueous solutions by plants. *J. Exp. Bot.* 17 (52), 569–578.
- Barboni, D., Bonnefille, R., Alexandre, A., Meunier, J.D., 1999. Phytoliths as paleo-environmental indicators, west side middle awash valley, Ethiopia. *Palaeogeogr. Palaeoclimatol. Palaeoecol.* 152, 87–100.
- Cabanes, D., Weiner, S., Shahack-Gross, R., 2011. Stability of phytoliths in the archaeological record: a dissolution study of modern and fossil phytoliths. *J. Archaeol. Sci.* 38, 2480–2490.

- Cabanes, D., Shahack-Gross, R., 2015. Understanding fossil phytolith preservation: the role of partial dissolution in paleoecology and Archaeology. *PLoS ONE* 10 (5), 1–16.
- Carr, G., 2009. Water Reuse for Irrigated Agriculture in Jordan (Unpublished Ph.D. thesis). Department of Soil Science, University of Reading, UK.
- Casey, W.H., Kinrade, S.D., Knight, T.G., Rains, D.W., Epstein, E., 2003. Aqueous silicate complexes in wheat, *Triticum aestivum* L. *Plant, Cell & Environ.* 27, 51–54.
- Charles, M., Hoppé, C., 2003. The effects of irrigation on the weed floras of winter cereal crops in Wadi ibn Hamad (southern Jordan). *Levant* 35, 213–230.
- Charles, M., Hoppé, C., Jones, G., Bogaard, A., Hodgson, J., 2003. Using weed functional attributes for the identification of irrigation regimes in Jordan. *J. Archaeol. Sci.* 30, 1429–1441.
- Crawley, M.J., 2007. *The R Book*. John Wiley & Sons, Chichester, UK.
- Ernst, W.H.O., Vis, R.D., Piccoli, F., 1995. Silicon in developing nuts of the sedge *Schoenus nigricans*. *J. Plant Physiology* 146, 481–488.
- Ferrio, J., Araus, J., Buxo, R., Voltas, J., Bort, J., 2005. Water management practices and climate in ancient agriculture: inferences from the stable isotope composition of archaeobotanical remains. *Veg. Hist. Archaeobotany* 14, 510–517.
- Finne, M., Norstrom, E., Risberg, J., Scott, L., 2010. Siliceous microfossils as late Quaternary paleo-environmental indicators at Braamhoek wetland, South Africa. *Holocene* 20, 747–760.
- Fiorentino, G., Caracuta, V., Calcagnile, L., D'Elia, M., Matthiae, P., Mavelli, F., Quarta, G., 2008. Third millennium B.C. Climate change in Syria highlighted by carbon stable isotope analysis of 14C-AMS dated plant remains from ebla. *Palaeogeogr. Palaeoclimatol. Palaeoecol.* 266, 51–58.
- Fiorentino, G., Caracuta, V., Casiello, G., Longobardi, F., Sacco, A., 2012. Studying ancient crop provenance: implications from  $\delta^{13}C$  and  $\delta^{15}N$  values of charred barley in a middle bronze age silo at ebla (NW Syria). *Rapid Commun. Mass Spectrom.* 26, 327–335.
- Flohr, P., Mülder, G., Jenkins, E., 2011. Carbon stable isotope analysis of cereals remains as a way to reconstruct water availability: preliminary results. *Water Hist.* 3, 121–144.
- Fraysse, F., Cantais, F., Pokrovsky, O.S., Schott, J., Meunier, J.D., 2006a. Aqueous reactivity of phytoliths and plant litter: physico-chemical constraints on terrestrial biogeochemical cycle of silicon. *J. Geochem. Explor.* 88, 202–205.
- Fraysse, F., Pokrovsky, O.S., Schott, J., Meunier, J.-D., 2006b. Surface properties, solubility and dissolution kinetics of bamboo phytoliths. *Geochimica Cosmochimica Acta* 70, 1939–1951.
- Fraysse, F., Pokrovsky, O.S., Schott, J., Meunier, J.-D., 2009. Surface chemistry and reactivity of plant phytoliths in aqueous solutions. *Chem. Geol.* 258, 197–206.
- Grömping, U., 2006. Relative importance for linear regression in R: the package relaimpo. *J. Stat. Softw.* 17 (1).
- Helback, H., 1960. Ecological effects of irrigation in ancient Mesopotamia. *Iraq* 22, 186–196.
- Heaton, T.H.E., Jones, G., Halstead, P., Tsipropoulos, T., 2009. Variations in the  $^{13}C/^{12}C$  ratios of modern wheat grain, and implications for interpreting data from bronze age assiros Toumba, Greece. *J. Archaeol. Sci.* 36 (10), 2224–2233.
- Hutton, J.T., Norrish, K., 1974. Silicon content of wheat husks in relation to water transpired. *Aust. J. Agric. Res.* 25, 203–212.
- Iler, R.K., 1979. *The Chemistry of Silica*. John Wiley and Sons, New York.
- Jarvis, S.C., 1987. The uptake and transport of silicon by perennial ryegrass and wheat. *Plant Soil* 97, 429–438.
- Jenkins, E., 2009. Phytolith taphonomy: a comparison of dry ashing and acid extraction on the breakdown of conjoined phytoliths formed in *Triticum durum*. *J. Archaeol. Sci.* 36, 2402–2407.
- Jenkins, E., Jamjoum, K., Al-Nuimat, S., 2011. Irrigation and phytolith formation: an experimental study. In: Mithen, S.J., Black, E. (Eds.), *Water, Life and Civilisation: Climate, Environment and Society in the Jordan Valley*. Cambridge University Press/UNESCO, Cambridge/New York, pp. 347–372.
- Jones, G., Charles, M., Colledge, S., Halstead, P., 1995. Towards the archaeobotanical recognition of winter-cereal irrigation: an investigation of modern weed ecology in northern Spain. In: Kroll, H., Pasternak, R. (Eds.), *Res Archaeobotanicae—9th Symposium IWGP*, Kiel. Institut für Ur- und Frühgeschichte der Christian-Albrecht-Universität.
- Jones, G., Charles, M., Bogaard, A., Hodgson, J., 2010. Crops and Weeds: the role of weed functional ecology in the identification of crop husbandry methods. *J. Archaeol. Sci.* 37, 70–77.
- Jones, L.H.P., Handreck, K.A., 1965. Studies of silica in the Oat plant. III. Uptake of silica from soils by the plant. *Plant Soil* 23, 79–96.
- Jones, L.H.P., Handreck, K.A., 1967. Silica in soils plants and animals. *Adv. Agron.* 19, 107–149.
- Jones, L.H.P., Milne, A.A., 1963. Studies of silica in the oat plant I. Chemical and physical properties of the silica. *Plant Soil* 18, 207–220.
- Jones, L.H.P., Milne, A.A., Wadham, S.M., 1963. Studies of silica in the Oat plant II. Distribution of silica in the plant. *Plant Soil* 18, 358–371.
- Liang, Y., Hua, H., Zhang, J., Cheng, C., Römhild, V., 2006. Importance of plant species and external silicon concentration to active silicon uptake and transport. *New Phytologist* 172 (1), 63–72.
- Loucaides, S., Van Cappellen, P., Behrends, T., 2008. Dissolution of biogenic silica from land to ocean: role of salinity and pH. *Limnol. Oceanogr.* 53, 1614–1621.
- Loucaides, S., Behrends, T., Van Cappellen, P., 2010. Reactivity of biogenic silica: surface versus bulk charge density. *Geochimica Cosmochimica Acta* 74, 517–530.
- Ma, J.F., Takahashi, E., 2002. *Soil, Fertilizer, and Plant Silicon Research in Japan*. Elsevier, Amsterdam-Boston-London-New York-Oxford-Paris-San Diego-San Francisco-Singapore-Sydney-Tokyo.
- Ma, J.F., Yamaji, N., 2006. Silicon uptake and accumulation in higher plants. *Trends Plant Sci.* 11 (8), 392–397.
- Mabry, J., Donaldson, M.L., Gruspier, K., et al., 1996. Early town development and water management in the Jordan valley: investigations at Tell el-Handaqq North. *Annu. Dep. Antiq. Jordan* 53, 115–154.
- Madella, M., Jones, M.K., Echlin, P., Powers-Jones, A., Moore, M., 2009. Plant water availability and analytical microscopy of phytoliths: implications for ancient irrigation in arid zones. *Quat. Int.* 193, 32–40.
- Mayland, H.F., Johnson, D.A., Asay, K.H., Read, J.J., 1993. Ash, carbon isotope discrimination, and silicon as estimators of transpiration efficiency in crested wheat-grass. *Aust. J. Plant Physiology* 20, 361–369.
- Mithen, S., Jenkins, E., Jamjoum, K., Nuimat, S., Nortcliff, S., Finlayson, B., 2008. Experimental crop growing in Jordan to develop methodology for the identification of ancient crop irrigation. *World Archaeol.* 40 (1), 7–25.
- Novello, A., Barboni, D., Berti-Equille, L., Mazur, J.-C., Poilecot, P., Vignaud, P., 2012. Phytolith signal of aquatic plants and soils in Chad, Central Africa. *Rev. Palaeobot. Palynology* 178, 43–58.
- R Core Team, 2013. *A Language and Environment for Statistical Computing*. R Foundation for Statistical Computing, Vienna, Austria. URL: <http://www.R-project.org/>.
- Rafi, M.M., Epstein, E., 1999. Silicon absorption by wheat (*Triticum aestivum* L.). *Plant Soil* 211, 223–230.
- Rambeau, C., Black, S., 2011. Palaeoenvironments of the southern Levant 5,000 BP to present: linking the geological and archaeological records. In: Mithen, S.J., Black, E. (Eds.), *Water, Life and Civilisation: Climate, Environment and Society in the Jordan Valley*. Cambridge University Press/UNESCO, Cambridge/New York, pp. 94–104.
- Richardson, A.E.V., Trumble, H.C., Shapter, R.E., 1931. Factors affecting the mineral content of pastures. *Counc. Sci. Industrial Res. Aust. (CSIR) Bull.* 49.
- Richmond, K.E., Sussman, M., 2003. Got Silicon? the non-essential beneficial plant nutrient. *Curr. Opin. Plant Biol.* 6, 268–272.
- Riehl, S., 2008. Climate and agriculture in the ancient near east: a synthesis of the archaeobotanical and stable carbon isotope evidence. *Veg. Hist. Archaeobotany* 17 (Suppl. 1), S43–S51.
- Riehl, S., Bryson, R., Pustovoytov, K., 2008. Changing growing conditions for crops during the near eastern bronze age (3000–1200 BC): the stable carbon isotope evidence. *J. Archaeol. Sci.* 35, 1–12.
- Robinson, S., Black, S., Selwood, B., Valdes, P.J., 2011. A review of palaeoclimates and palaeoenvironments in the Levant and Eastern Mediterranean from 25,000 to 5,000 years BP: setting the environmental background for the evolution of human civilisation. In: Mithen, S.J., Black, E. (Eds.), *Water, Life and Civilisation: Climate, Environment and Society in the Jordan Valley*. Cambridge University Press/UNESCO, Cambridge/New York, pp. 71–93.
- Rosen, A.M., Weiner, S., 1994. Identifying ancient irrigation: a new method using opaline phytoliths from emmer wheat. *J. Archaeol. Sci.* 21, 125–132.
- Stokes, H., Mülder, G., Jenkins, E., 2011. An investigation into the archaeological application of carbon stable isotope analysis used to establish crop water availability: solutions and ways forward. In: Mithen, S.J., Black, E. (Eds.), *Water, Life and Civilisation: Climate, Environment and Society in the Jordan Valley*. Cambridge University Press/UNESCO, Cambridge/New York, pp. 373–380.
- Takahashi, E., Ma, J.F., Miyake, Y., 1990. The possibility of silicon as an essential element for higher plants. *Comments Agric. Food Chem.* 2, 357–360.
- Tsartsidou, G., Lev-Yadun, S., Albert, R.-M., Miller-Rosen, A., Efratiou, N., Weiner, S., 2007. The phytolith archaeological record: strengths and weaknesses evaluated based on a quantitative modern reference collection from Greece. *J. Archaeol. Sci.* 34, 1262–1275.
- Underwood, A.J., 1997. *Experiments in Ecology: Their Logical Design and Interpretation Using Analysis of Variance*. Cambridge University Press, Cambridge.
- Van Der Vorm, P.D.J., 1980. Uptake of Si by five plant species, as influenced by variation in Si-supply. *Plant Soil* 56, 153–156.
- Voltas, J., Ferrio, J., Alonso, N., Araus, J., 2008. Stable carbon isotopes in archaeobotanical remains and palaeoclimate. *Contributions Sci.* 4 (1), 21–31.
- Walker, C.D., Lance, R.C.M., 1991. Silicon accumulation and  $^{13}C$  composition as indices of water-use efficiency in barley cultivars. *Aust. J. Plant Physiology* 18, 427–434.
- Wallace, M., Jones, G., Charles, M., Fraser, Halstead, P., Heaton, T.H.E., Bogaard, A., 2013. Stable carbon isotope analysis as a direct means of inferring crop water status and water management practices. *World Archaeol.* 45.3, 388–409.
- Wallace, M.P., Jones, G., Charles, M., Fraser, R., Heaton, T.H.E., Bogaard, A., 2015. Stable carbon isotope evidence for Neolithic and bronze age crop water management in the Eastern Mediterranean and Southwest Asia. *PLoS ONE* 10 (6).
- Weisskopf, A., Qin, L., Ding, J., Ding, P., Sun, G., Fuller, D., 2014. Phytoliths and rice: from wet to dry and back again in the Neolithic Lower Yangtze. *Antiquity* 89, 1051–1063.